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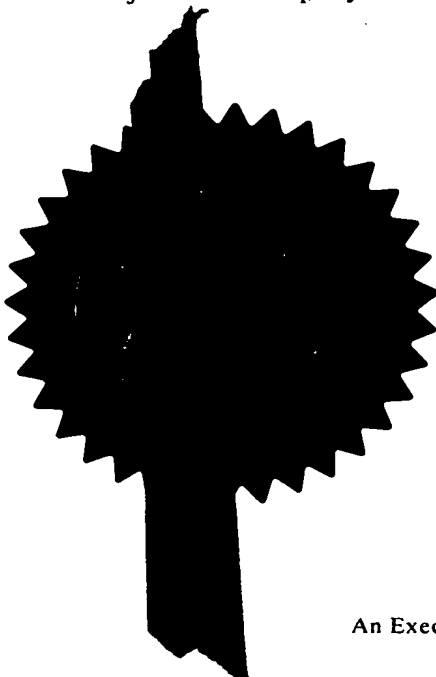
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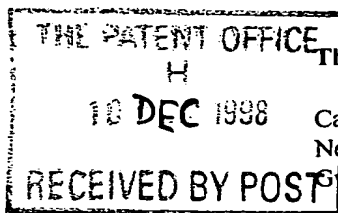
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2.	Patent application number (The Patent Office)	<div style="text-align: right;">10 DEC 1998</div> <div style="text-align: center; font-size: 1.5em; font-weight: bold;">9827033.3</div>	
3.	Full name, address and postcode of the or of each applicant ( <u>underline all surnames</u> )	<div style="text-align: right;">KEMGAS LIMITED The Corner House, Parliament Street, Hamilton, Bermuda.</div>	
	Patents ADP number ( <i>if you know it</i> )		
	If the applicant is a corporate body, give the country/state of its incorporation	<div style="text-align: right;">Bermuda <span style="font-size: 1.5em; font-family: cursive;">7565955001</span></div>	
4.	Title of the invention	LIME TREATMENT	
5.	Name of your agent ( <i>if you have one</i> )	Marks & Clerk	
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	Patents ADP number ( <i>if you know it</i> )	<div style="text-align: right;">18004</div> <div style="text-align: right; font-size: 2em;">✓</div>	
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Signature

*M. Atkinson*  
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Date 9 December 1998

12. Name and daytime telephone number of person to contact in the United Kingdom

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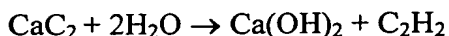
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### Lime Treatment

The present invention relates to a method of treating lime and more particularly, but not exclusively, to the treatment of lime containing insoluble impurities, to obtain a purified solution of calcium ions and also the use of said processing method to obtain useful, solid calcium containing products from the lime. The invention relates more particularly, but again not exclusively, to the treatment of carbide lime.

Carbide lime is a by-product in the production of acetylene by the reaction of calcium carbide and water according to the equation



More particularly, carbide lime is comprised of calcium hydroxide and impurities resulting from the original calcium carbide and possibly also from the conditions under which the acetylene is produced.

The carbide lime is produced in amounts of approximately 3.5-4 times the weight of acetylene and is produced in dry powder form coming from a dry gas generator but mostly it is a water slurry from wet generators. Carbide lime is also known as carbide sludge, generator slurry, lime sludge, lime hydrate, and hydrated carbide lime.

Carbide lime is a grey-black substance. Typically it consists of around 90% by weight of calcium hydroxide (based upon the solids content of the carbide lime), the remainder being impurities which depend upon the method used to manufacture the acetylene and also upon the source of the materials used to manufacture the calcium carbide (normally made by roasting calcium oxide and coal). The main impurities are the oxides of silicon, iron, aluminium, magnesium, and manganese combined with carbon, ferrosilicon and calcium sulphate. Additionally if the carbide

lime is stored outside, calcium carbonate, formed by the reaction of calcium hydroxide with carbon dioxide, may be present as an impurity.

Due to the impurities present in the carbide lime it has a low market value and is difficult to sell. The limited number of uses include use as a cheap base to neutralise acids or use in a slightly modified form as an agricultural fertiliser (Czechoslovakian Patent Application CS 8002961 – Jansky).

Since it does not have a significant commercial use, and also because the impurities which it contains renders disposal difficult, there are millions of tonnes of carbide lime stored in carbide lime pits all over the world. These pits are an ever increasing environmental problem.

Several methods as outlined below have been proposed to purify carbide lime but have various disadvantages:-

- a) Heating. The water and carbon impurities in the carbide lime can be removed by heating carbide lime in an oven at a temperature of at least  $800^{\circ}\text{C}$  to yield a "white" lime. However, this process is expensive to operate and has the disadvantage that oxide impurities are not removed.
- b) Simple filtration. The sludge may be subjected to a filtration operation. Unfortunately the particle size of the impurities contained within the carbide lime is similar to that of the particles of calcium hydroxide at  $1\text{ }\mu\text{m}$  to  $50\text{ }\mu\text{m}$ . Also as the impurities in the carbide lime tend to block filters with a thick sludge, the filters suffer from reduced efficacy and need constant replacement. Therefore simple filtration is ineffective.
- c) Dissolution of calcium hydroxide in water followed by filtration. As calcium hydroxide is sparingly soluble in water and as most of the impurities in carbide lime are insoluble, the calcium hydroxide can be extracted into an aqueous solution which is then filtered to remove the



impurities. Unfortunately calcium hydroxide is only sparingly soluble in water; some 650 cubic metres of water are needed to dissolve one tonne of calcium hydroxide, therefore this method is impractical industrially.

- d) Solution of calcium hydroxide in water, using an ammonium salt as a solvating aid, followed by filtration. This method is identical to that described in (c) with the exception that anions, provided as chloride or the nitrate, are used to increase the solubility of the calcium hydroxide in water. This method is effective in reducing the amount of water needed to dissolve the calcium hydroxide but suffers from the drawback that the liquor containing the ammonium poses an effluent problem due to the relatively high ammonium salt concentrations, unless the ammonium solution is recycled after precipitation of the calcium with carbon dioxide.

The difficulties involved in purifying carbide lime means that, in spite of the large available amounts of this material, it is not used as a source of calcium for the production of higher value calcium products which have significant industrial applications. One example of such a product is Precipitated Calcium Carbonate (PCC) which is used as a functional filler in materials such as; paints, paper, coatings, plastics, sealants and toothpaste.

PCC is currently manufactured by the following methods: -

- a) Reacting an aqueous slurry of lime with carbon dioxide. This method suffers from the disadvantage that it is slow due to the low solubility of lime.
- b) Reacting a solution of lime with carbon dioxide. In this case problems arise initially as lime is only sparingly soluble in water (typical saturated concentration  $2.16 \times 10^{-2}$  molar at room temperature). The low concentration presents separation problems once the conversion to PCC is complete. Also due to the low lime concentration the reaction equilibria

are such that, in the reaction with carbon dioxide, only about 30% of the lime is converted to PCC, the remainder being converted to  $\text{Ca}(\text{HCO}_3)_2$ , which remains in solution.

It is therefore an object of the present invention to obviate or mitigate the above mentioned disadvantages.

According to a first aspect of the present invention there is provided a method of obtaining a solution of calcium ions from lime containing insoluble impurities, comprising

- (i) treating the lime with an aqueous solution of a polyhydroxy compound to extract calcium from the lime, and
- (ii) separating insoluble impurities from the solution resulting from (i)

The lime used in the method of the invention may be any lime containing impurities that are insoluble in an aqueous solution of a polyhydroxy compound. A preferred example of such a lime is carbide lime, which contains carbon, ferrosilicon, calcium sulphate and the oxides of iron, silicon, aluminium, magnesium and manganese as insoluble impurities.

The polyhydroxy compound solution acts as a solvent for the calcium and allows a much higher amount (e.g. about 65g/l) of the calcium ions present in the lime to go into solution than would be the case of use of only water. The method of the invention provides an efficient procedure for extracting calcium from lime. After removal of insoluble impurities, there remains a purified solution of calcium ions which may be used for the production of calcium containing products of considerably higher commercial value than carbide lime as discussed more fully below.

The polyhydroxyl compound used in the method of the invention should be one having significant solubility in water under the conditions employed and may for

example be a polyhydric alcohol, a saccharide (e.g. a mono- or di- saccharide) or a saccharide derivative. Examples of polyhydric alcohols which may be used include tri-hydric and higher functionality alcohols, e.g. glycerol, butanetriol, trimethylolpropane, or erythritol.

Examples of saccharides which are useful in the invention include glucose, fructose, ribose, xylose, arabinose, galactose, mannose, sucrose, lactose and maltose. Examples of saccharide derivatives which are useful in the invention include saccharide alcohols such as sorbitol and mannitol. It is particularly preferred that the polyhydroxyl compound is chosen from the group consisting of sucrose, glucose, sorbitol and glycerol.

Most preferably the polyhydroxy compound is sorbitol. The use of sorbitol is an important feature of the invention in its own right and therefore according to a second aspect of the invention there is provided a method of obtaining a solution of calcium ions from lime comprising treating the lime with an aqueous solution of sorbitol to extract calcium from the lime.

The second aspect is applicable whether or not the lime contains insoluble impurities. For example the second aspect of the invention may be used to obtain a solution of  $\text{Ca}^{2+}$  ions from relatively pure lime.

Depending on its solubility in water at the temperature used in the method, the polyhydroxy compound will generally be employed as a 10% to 80% by weight solution in water. When the polyhydroxyl compound is a saccharide, or a derivative thereof, it will generally be used as 10% to 60% by weight solution, more preferably 15% to 40% by weight solution in water. In contrast, glycerol will generally be used as 60% to 80% by weight solution in water more preferably 65% to 75% by weight solution.

The invention will be described fully with reference to the treatment of carbide lime but it is applicable *mutatis mutandis* to other forms of lime.

To produce a purified solution of calcium ions from carbide lime, it would generally be appropriate to extract an amount of carbide lime providing 3 to 12, more preferably 3 to 7 and ideally about 5 parts by weight calcium hydroxide with 100 parts by weight of the aqueous solution of the polyhydroxyl compound. Dry carbide lime from an acetylene generator may be extracted without further processing. However, in the case of wet carbide lime it will generally be preferred that this will be allowed to settle and subsequently dewatered prior to the extraction step. This can be best be done by filtration.

The extraction step is preferably effected at a temperature of 5°C to 60°C, which has the advantage of preventing thermal decomposition of the polyhydroxyl compound although we do not preclude the use of temperatures outside this range. The admixture of the carbide lime and aqueous solution of the hydroxy compound should also be agitated to ensure maximum extraction of calcium ions into the aqueous liquor. Treatment times to obtain a desired degree of extraction will depend on factors such as the temperature at which the extraction is performed, degree of agitation, and concentration of the polyhydroxy compound but can readily be determined by a person skilled in the art.

Subsequent to the extraction step, the calcium ion solution is separated from insoluble impurities. Conveniently separation is effected by filtration, e.g. using a microfiltration unit, but other methods may be employed.

The resultant product is a purified calcium ion containing solution which may be used, for example, as a feedstock for producing industrially useful calcium containing, solid products. Such products are most conveniently produced by a precipitation reaction in which a chemical agent is added to the solution to precipitate the desired product. Thus, for example, by bubbling carbon dioxide through the purified calcium ion containing solution it is possible to produce Precipitated Calcium Carbonate. Other precipitating agents which may be used include phosphoric acid, sulphuric acid, oxalic acid, hydrofluoric acid and citric acid.

Generally it will be appropriate to add the precipitating agent in a stoichiometric amount to the calcium contained in the solution. Alternatively or additionally, the supernatant liquor remaining after the precipitation reaction may be recycled for use in extracting calcium from a fresh batch of carbide lime. If the supernatant is to be recycled then it is desirable to dewater the carbide lime to prevent too much water entering the recycle stream and undesirably diluting this solution of the polyhydroxyl compound. Thus, as indicated previously, if the wet carbide lime is to be treated it should be allowed to settle and then dewatered.

For the production of Precipitated Calcium Carbonate, carbon dioxide may be bubbled through the purified calcium ion solution using a conventional carbonation reactor. This reaction may be conducted at ambient temperature. Additives to coat the PCC; e.g. stearic acid derivatives, may be added at a later stage if required.

The PCC may be dewatered, washed and dried using equipment well known in the art.

The particle size of the PCC produced will depend upon parameters such as reaction time, temperature, CO<sub>2</sub> concentration and agitation speed.

The described method of producing PCC has the following advantages.

1. The method allows the production of high purity PCC.
2. The calcium ions, from which the calcium carbonate is generated are present in solution at a much higher concentration than would be the case of treating a suspension of lime.
3. Compared to the use of a suspension of lime for generating PCC, the method of the invention does not result in PCC being "deposited" on lime particles.
4. The method of the invention yields a PCC of narrow size distribution, small particle size and good colour.

The invention will be further illustrated by the following, non-limiting Examples

**Example 1**

57.5 grams of carbide lime, containing approximately 7.5 grams of impurities such as calcium carbonate, oxides and sulphates of silicon, iron, aluminium, magnesium, and manganese with carbon and ferrosilicon, were dissolved with stirring for 15 minutes in a solution containing 250 grams of sucrose in 750 grams of water.

The resulting solution containing undissolved black covered sludge-like impurities was filtered.

The clean solution containing about 50grams purified calcium hydroxide was transferred to a carbonation reactor for precipitation of calcium carbonate by reaction with carbon dioxide gas.

After 10-20 minutes of bubbling carbon dioxide the reaction to calcium carbonate was complete. The precipitated calcium carbonate (PCC) was filtered out of the suspension and dried, yielding 67 grams of calcium carbonate, (expected ~67.5 grams, implying a yield of over 99%).

The PCC powder had the following properties

Mean Particle Size	3 $\mu$ m
Crystal Structure	Calcite Rhombic Structure
Acid Insolubles	<0.06%
Residual Fe	<3 ppm
Residual Mg	<3 ppm
Residual Mn	<3 ppm
Residual S	<3 ppm
Residual Al	33 ppm
Residual Silica	300 ppm

### Example 2

To a 2 litre round bottomed flask fitted with a mechanical stirrer and thermometer was charged 250 grams of sorbitol in 660 grams of water at ambient temperature. To the resultant clear solution was charged 100 grams of crude carbide lime containing 50% moisture. The mixture was then stirred for a minimum of 20 minutes.

When the resulting solution containing the undissolved impurities was filtered the clear filtrate obtained was found to contain 4.1% w/w calcium hydroxide. The filtrate was charged to a carbonation reactor for precipitation of calcium carbonate by reaction with carbon dioxide gas, using the method described below.

To a 2 litre round bottomed flask fitted with a mechanical stirrer, pH probe and gas sparge tube was charged 4% calcium hydroxide in sorbitol solution (1000 grams). After sparging the mixture with carbon dioxide for approximately 10 minutes the reaction to calcium carbonate was complete, indicated by the pH change from 11.8 to 7.0.

The precipitated calcium carbonate (PCC) was filtered and dried to yield 54.2 grams of calcium carbonate. The theoretical yield is 55.1 grams, implying a yield of 98.3%.

The fine, white PCC powder had the following properties.

	PCC	Carbide Lime
Mean Particle Size	~2 $\mu$ m	1-50 $\mu$ m
Crystal Structure	Calcite Rhombic Structure	-
Acid Insolubles	<0.2%	
Residual Fe	<0.05%	0.12%
Residual Mg	<0.05%	0.07%
Residual S	<0.1%	0.35%
Residual Al	<0.05%	1.15%
Residual Silica	<0.1%	1.5%



Claims

1. A method of obtaining a solution of calcium ions from lime containing insoluble impurities, comprising
  - (i) treating the lime with an aqueous solution of a polyhydroxy compound to extract calcium from the lime, and
  - (ii) separating insoluble impurities from the solution resulting from (i).
2. A method according to claim 1 wherein the lime is carbide lime.
3. A method according to claim 1 or 2 wherein the polyhydroxy compound is a polyhydric alcohol, a saccharide or a saccharide derivative.
4. A method as claimed in claim 3 wherein the polyhydroxy compound is glucose, fructose, ribose, xylose, arabinose, galactose, mannose, sucrose, lactose or maltose.
5. A method as claimed in claim 3 wherein the saccharide derivative is sorbitol or mannitol.
6. A method as claimed in claim 3 wherein the polyhydroxy compound is a tri-hydric or higher functionality alcohol.
7. A method as claimed in claim 6 wherein the polyhydroxy compound is glycerol, butanetriol, trimethylolpropane or erythritol.
8. A method as claimed in any one of claims 1 to 7 wherein the polyhydroxy compound is employed as 10% to 80% by weight solution in water.
9. A method as claimed in claim 8 wherein the polyhydroxy compound is a saccharide or derivative thereof and is employed as a 10% to 60% by weight solution.

10. A method as claimed in claim 8 wherein the polyhydroxy compound is glycerol and is employed as 60% to 80% by weight solution water.

11. A method as claimed in any one of claims 1 to 10 wherein the amount of carbide lime is such as to provide 3 to 12 parts by weight per 10 to 80% by weight of the polyhydroxy compound.

12. A method as claimed in any one of claims 1 to 11 effected at a temperature of 5°C to 60°C.

13. A method of obtaining a solution of calcium ions from lime comprising treating the lime with an aqueous solution of sorbitol to extract calcium from the lime.

14. A method of producing a calcium containing product comprising the steps of

(i) preparing a solution of calcium ion according to the procedure of any one of claims 1 to 13, and

(ii) adding to the solution from (i) a precipitating agent which cause precipitation of the desired calcium containing product.

15. A method as claimed in claim 14 wherein the precipitating agent is carbon dioxide and the product obtained is precipitated calcium carbonate.

16. A method of producing precipitated calcium carbonate from carbide lime comprising

(i) treating the carbide lime with an aqueous solution of a polyhydroxy compound to extract calcium from the carbide lime,

(ii) separating insoluble impurities from the solution resulting from (i), and

(iii) treating the solution with carbon dioxide.